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Sample Accountability Quality Assurance for the 'Integrated Air Cancer Project' Research Program of the U.S. Environmental Protection Agency

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16. ABSTRACT

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CHEMICAL CONTAMINANTS MONITORING

Sample Accountability Quality Assurance for the "Integrated Air Cancer Project" Research Program of the U.S. Environmental Protection Agency

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A sample accountability quality assurance (QA) program is described for a field and laboratory research effort which resulted in collection of approximately 2000 samples for analysis by several EPA and contractor laboratories. A QA program was specifically developed for this research program to include sample transfer from collection site to storage maintenance, record development, transfer to researchers, and sample tracking at all stages. A sample identification system and sample custody records are described for field and laboratory application. The functions of a sample coordinator are also described as relating to sample custody, coordination of sample analysis with researchers, and development of computer record files to facilitate research and sample tracking.

The Integrated Air Cancer Project (IACP) of the U.S. Environmental Protection Agency (EPA) was established in 1984 as a multiyear research program in response to the Agency's mandate for regulating the emission of carcinogens into the air. Past research on hazardous air pollutants was generally conducted on individual primary pollutants at sites selected for specific reasons. The IACP has adopted a different approach of monitoring ambient residential neighborhood air for all chemical species likely to be mutagenic and/or carcinogenic. The goals of the project are to (*1*) identify the principal airborne carcinogens, (*2*) determine which emission sources are the major contributors of carcinogens to ambient air, and (*3*) improve the estimate of comparative human cancer risk from specific pollution emission sources. Meeting these goals required the IACP planners to bring together the multidisciplinary talents of chemists, engineers, and health scientists to conduct a series of coordinated field studies of emission sources and of atmospheric transport and transformation and toxicological studies to address the complex issues involved. This mix of scientific capability was accomplished by combining expertise from the 4 EPA Office of Research and Development laboratories at Research Triangle Park, NC.

Phase 1 (1985) of the sampling program of the IACP studied simplified residential air sheds in 2 neighborhoods heavily impacted by woodsmoke. The objectives of this first phase were to (*1*) identify and quantitate carcinogens in ambient air resulting from residential wood combustion and motor vehicles, (*2*) quantitate the relative contributions of specific emission sources to the mutagenic activity, organic, and fine particulate mass of ambient airborne pollutants, and (*3*) develop and test exposure assessment methodology for selected carcinogens. The diversified goals of this phase 1 study resulted in 25 research projects and collection of about 2000 air samples.

The complexity of coordinating this large sampling effort with the numerous researchers and research tasks involved was recognized during study planning. Sample tracking and coordination was a particular concern because 2 or more laboratories were often expected to perform different functions on the same sample, i.e., extraction, determination of total extractable organic matter, analytical fractionation, characterization, and bioassay. The study planners therefore directed the development of a sample accountability quality assurance (QA) program. Detailed procedures were to be established for storage and transfer of samples and related documentation so that strict integrity of the study would be maintained.

A committee was appointed to establish procedures, develop forms, and designate key personnel to institute a QA program for sample transfer from collection site to storage maintenance, record development, transfer to researchers, and sample tracking at all stages. Quality assurance plans were also adopted for management of the field sampling program but will not be discussed here.

Data management was also of primary concern. Research findings would be forthcoming from the various laboratories with data that would often apply to the same samples or sample sets. A central data management office was therefore established to receive copies of all sample records coming from the field sampling site as well as research results from individual laboratories and researchers.

The purpose of this paper is to relate the QA procedures developed and information gained from this first phase of the IACP and to make recommendations relevant to QA plans for similar field sampling and research programs. The procedures described proved adequate and are recommended for research programs involving a number of research tasks and many field and laboratory personnel. Not all aspects of this QA plan, however, would be appropriate for regulatory investigations. An excellent review of other forms and applications of QA programs may be found in an AOAC publication on quality assurance (Garfield, F. M. [1984] *Quality Assurance Principles for Analytical Laboratories*, AOAC, Arlington VA).

Sample Identification

The starting point of this sample accountability QA program was the development of a sample description/identification form that would be used at the field sampling sites. An example of the subsequently developed form is shown in Figure 1. The form provides 2 types of sample information. The top portion relates all pertinent sample collection information, and the lower portion is devoted to assignment of an alphanumeric sample identification number.

The sample numbering system was given careful consideration because several hundred samples would be generated in the field, and the same sample might be subject to analytical operations in 2 or more laboratories. The system needed to be simple for field use and flexible enough to reflect subsequent analytical manipulations as later discussed. A sample numbering system was developed which would (*1*) identify the sample program, year, and collection site, (*2*)

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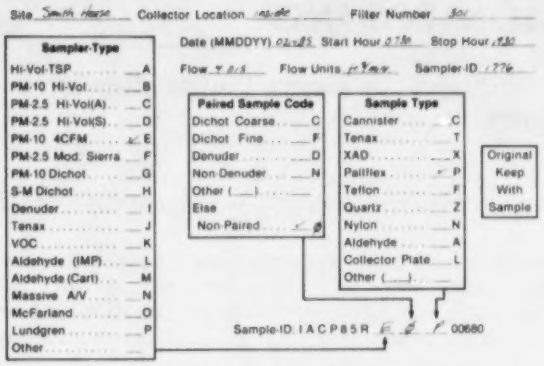

Figure 1. Sample identification form.

relate descriptive information about the sample, and (*3*) contain a distinguishing 5 digit number. The sample form as supplied to the field contained a preassigned sample identification number with the exception of the 3 descriptor spaces as later described, e.g., IACP85R_____00680 (see Figure 1). This simple precaution of supplying forms with serialized identification numbers precluded the possibility of the sampling team issuing duplicate numbers. The IACP first portion of the preprinted identification number represented the Integrated Air Cancer Project and 85R designated the 1985 sampling program in Raleigh, NC. The next 3 letter characters related the sample descriptive information and were to be assigned by sampling team personnel. The first space, selected from the field indicated on the form in Figure 1, identified the sampler type. The second letter entry, selected from the "paired sample code" field, distinguished between 2 samples collected simultaneously by the same sampler, e.g., C and F entries for a dichot sampler coarse and fine filters. The third space entry identified the sample medium used.

Assignment in the Field

Instructions accompanying the forms to the field gave directions for special case situations as follows: (*1*) sample blanks be indicated by writing "blank" on the "other" line in the "sampler-type" field and a "Z" letter code entered which would also become the first letter of the 3 space identification code number; (*2*) samplers not listed in the sampler-type field would be written in on the "other" line and a "Y" letter code also entered which would become the first letter of the 3 space identification code number; and (*3*) sample types not listed would be described on the "other" line of the third field and a "Y" letter code entered in the third space.

A quantity of the preserialized identification forms were prepared as 2 part duplicates and supplied to the sampling site along with detailed instructions for their use. After the form was completed, it was separated at the perforation and one portion was transmitted to the central data management office; the duplicate portion was kept with the sample for subsequent transport and receipt by the laboratory sample custodian.

Laboratory Modifications/Expansions

Instructions for the sample identification system also included special case situations for the analytical laboratory as follows: (*1*) blank samples generated from the laboratory would be accompanied by a new sample identification form with the first of the 3 descriptor letters again designated with a "Z"; and (*2*) samples or extracts that were *composited* in the laboratory would also have a new identification form prepared with a "Z" designation in the middle, or "paired sample code," field. The new identification form would also contain a listing of parent samples used to make up the composite.

Two other analytical operations were considered which affected the sample identification number, namely, the use and transfer of aliquots and the creation of additional subsamples through some analytical fractionation scheme. Since these operations did not change the accuracy of the information associated with the original sample, new identification numbers were not required. Instead, these operations were indicated by suffixes added to the original sample identification number. The suffix rules adopted directed alternate aliquot field, fractionation field, aliquot field, fractionation field, etc. Specific format was as follows.

Aliquot field.—This was denoted by 3 characters, Ann, where "A" is a mnemonic for "Aliquot" and "nn" was a 2 digit number. The bulk sample was assumed to be A00. Even if no aliquots were taken and the whole sample were used for fractionation, the Sample ID suffix would be written ID#-A00-FXnn.

Fractionation field.—This field consisted of 4 characters, FXnn, where "F" represented "Fractionation," "X" was a letter code for the type of fractionation, and "nn" was a 2 digit number. The letter codes for the type of fractionation were: N—normal phase fraction, R—reverse phase fraction, and G—silica gel fraction. The 2 digit number represented the various "cuts" from the fractionation. "Cut" points were to be documented in the laboratory's internal records. The use of the number 00 had a special meaning. If no fractionation was actually performed, but an aliquot was taken from a previous aliquot, then the suffix "FX00" was used as a "place keeper" to separate the aliquot fields. For example, a sample identification number with multiple aliquots and/or fractionations would take the form:

IACP85RXXXnnnnn-Ann-FXnn-Ann-FXnn.

where "X" represents an alphanumeric character and "n" represents an appropriate digit. Note that the aliquot fields and the fractionation fields must alternate.

Sample Accountability

The IACP Steering Committee developed overall goals for the sample accountability program from sampling site storage and maintenance, transport and transfer to the laboratory sample custodian, transfer to individual researchers, and sample tracking/record keeping. Although this first phase of the IACP was not directly related to a regulatory activity, the committee determined that sample custody procedures would be used and that one person should be designated as a sample coordinator. This person was responsible for sample custody and tracking from the field sampling site through all analytical operations. Collected samples were properly labeled and immediately stored at the sampling site under cryogenic conditions (−80°C). Sample transfers from the collection sites were made with samples contained in a cooler packed with dry ice. Sample custody forms and identification forms for all transferred samples were included with samples received by the laboratory sample custodian. Samples were immediately stored in a cryogenic freezer subsequent to distribution to individual researchers.

Sample Custody and Transfer

All sample transfers were recorded on a custody form as shown in Figure 2. A "pedigree" file consisting of at least a

Integrated Air Cancer Project
Sample Custody Form

Sample ID No.					
Description:					
Released by	Received by	Date	Time	Reason for Transfer	Storage and/or Transfer Conditions
Comments: (Parent ID No., etc.)					

Figure 2. Sample custody form.

IACP Sample Operations Record Page____

Sample ID No.	Sample Description	Amount of Original Sample	Date Received	Received by

Date	Analyst and Organization	Sample Analysis Procedure	Amount of Sample Consumed	Amount of Sample Remaining	Result or Data	Storage Location or Disposition

Figure 3. Sample operations record.

sample identification form and a custody record accompanied each sample during transfer. One copy of the signed custody form was forwarded to the sample custodian, a second copy was retained by the person releasing the sample, and the original form was transferred with the sample to the laboratory analyst.

Sample Operations Record

The "pedigree" file transferred with the sample could also contain a sample operations record which detailed preceding laboratory manipulations or measurements. A copy of a suggested operations record is shown in Figure 3. This particular format was not mandatory because laboratories usually had their own specialized forms which better reflected their particular operations.

Sample Coordination

The sample coordinator was responsible for 3 primary functions: sample custody, tracking all sample operations from collection site through the research tasks, and working with researchers to develop and assemble specific sets of samples required for special research tasks. A sample tracking computer file that used a Lotus software program was developed by the sample custodian. An excerpted portion of the file is shown in Figure 4. This file represents the total inventory of samples available and also relates pertinent sample collection parameters, i.e., sampler flow rate, total collection time, and fine particle mass concentrations for that time period. This added information gave invaluable assistance to analysis planning since samples could be ordered by concentration or amount collected. The file also contained sample custody information with fields for transfer date, received by, and reason for transfer. This portion of the file was periodically updated as custody sheets were received for processing. Fields were chosen to allow maximum flexibility in sorting or ordering samples by various parameters such as the 5 digit portion of the identification number, the 3 letter sample descriptor numerics from the identification number, collection date, individuals releasing or receiving samples, transfer dates, laboratory operations, or sample site. A description or notes field was also included to give added flexibility to sample records.

Additional computer files were also prepared for specific research tasks that required compilation of special sets of samples. For example, a residential sampling portion of the program required development of a file to show matched samples collected inside homes with samples from like collectors immediately outside the home and at the primary collection site located some blocks away. The sample coordinator assembled these samples as laboratory investigational units and coordinated their analyses with the various laboratories involved.

Conclusion and Recommendations

This first phase of the IACP provided valuable experience regarding sample accountability QA for a large field and laboratory research program. The QA procedures and formats described here proved adequate; however, they were not completely instituted until after the field sampling had begun. This initial delay caused some confusion for both field and laboratory personnel during the early part of the program. This phase 1 effort, however, was intended to be a learning experience in combining expertise and coordinating the work of 4 separate laboratories within EPA.

From the experience gained in the first phase of the IACP, some general thoughts and specific recommendations might now be made for future studies. QA considerations for sample handling, coordination, and tracking should be of primary importance to program planners. A sample coordinator should be appointed during preliminary planning. A committee consisting of both field sampling and laboratory personnel should also be established in early phases of planning to ensure development and finalizing of sample accountability procedures far in advance of actual field work. These QA considerations should include sample labeling and storage at field site, development and use of sample identification forms and custody forms for sample transfer, sample transfer conditions, receipt and storage of samples by the laboratory sample custodian, sample coordination and tracking, and data management.

Specific recommendations resulting from this study relate to sample coordination and use of the sample identification form which also contains the vital sampling parameter information. The 2 part duplicate form as used in this study led to a few copying errors. This form should consist of an

Integrated Air Cancer Project—Raleigh, NC, 1985 (IACP85R...)
Sample Inventory and Disposition

Date Coll'd	IACP Code	IACP#	Filter#	AM PM	Site	Flow m^3/min	Total Min	Fine $\mu g/m^3$	T'fer Date	Rec'd by	Reason	Notes
850119	COP	00567	2	PM	PRI	1.088	789.5	78	860400	R. Williams	Extraction	Alaska Biostudy 3
850119	COP	00563	5	PM	PRI	1.357	789.6	78				
850119	COP	00568	6	PM	PRI	1.217	789.5	78	851224	R. Williams	Task 20	Task 20, Follow-up Study
850119	COP	00566	3	PM	PRI	1.322	789.6	78				

Figure 4. Lotus file: Sample inventory and disposition.

original and 2 carbon copies to be completed by field personnel. The original should always be kept with the sample and one copy should be retained by the sample coordinator. The remaining copy would be forwarded to the data management staff. The sample coordinator should develop and continually update a sample inventory/custody record computer file. It is necessary for the sample coordinator to have an active part in planning the sample accountability portion of the QA plan and also be an active participant in specific research tasks requiring development of special sample sets and interlaboratory coordination for the various aspects of sample analysis.

The sample identification number expansion system for identification of analytical fractions was found to be inflexible since only forms of column chromatography were considered. An improved version would account for other fractionation techniques, e.g., liquid/liquid partitioning. An "O" letter for "other" could be used to indicate a fractionation technique other than those specifically mentioned.

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